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ABSTRACT

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Results of particulate contamination analyses by the Millipore and Whatman methods have been correlated for samples representing a wide range of contamination levels. These data indicate that although the different types of filters afford approximately equal particle retention, the modes of retention are markedly different. The particles are retained almost exclusively on the surface of the Millipore filter; whereas, with the Whatman filter, some particles are retained on the surface and others are embedded in the filter structure. There also appears to be a significant difference in the ease with which particles retained on the surfaces of these different types of filters can be distinguished microscopically. It, therefore, appears that any numerical equation relating the results obtained by these methods must be restricted to the particular type of contaminant present in the samples for which the equation was derived.

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J. O. Romine and J. B. Gayle

MATERIALS DIVISION
PROPULSION AND VEHICLE ENGINEERING LABORATORY

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SUMMARY

Results of particulate contamination analyses by the Millipore and Whatman methods have been correlated for samples representing a wide range of contamination levels. These data indicate that although the different types of filters afford approximately equal particle retention, the modes of retention are markedly different. The particles are retained almost exclusively on the surface of the Millipore filter; whereas, with the Whatman filter, some particles are retained on the surface and others are embedded in the filter structure. There also appears to be a significant difference in the ease with which particles retained on the surfaces of these different types of filters can be distinguished microscopically. It, therefore, appears that any numerical equation relating the results obtained by these methods must be restricted to the particular type of contaminant present in the samples for which the equation was derived.

INTRODUCTION

The method for determining the level of particulate contamination for most surfaces and components at this Center involves filtering a sample of fluid rinsings from the surface or component through a Whatman #42 analytical filter paper and microscopically sizing and counting the particulate material retained on the filter surface. This method is known to give only relative results, although it has been used extensively as a "quick check" procedure for contamination control of cleaning processes for components and systems.

A similar procedure, involving use of a membrane type (Millipore) filter, has been developed as an "absolute" reference method for particulate contamination analyses of fluids used in hydraulic and pneumatic systems. Application of this procedure to the control of contamination for cleaning of surfaces and components is suggested frequently and appears logical since no appreciable increase in the cost per determination would be involved. However, before making such change, it is necessary to determine acceptable contamination levels for the Millipore method compatible with those already established for the Whatman method. Therefore, the purpose of this investigation was to study the correlation of results obtained by the two methods and to consider various factors influencing this correlation.

EXPERIMENTAL

A total of seven contamination levels was studied, the first four with a laboratory recirculating system and the last three with a single pass pressurized system.

The laboratory system consisted of a centrifugal pump, a five gallon reservoir, associated plumbing, and a sampling valve located downstream from the pump. Prior to each series of tests, the fluid was circulated for at least thirty minutes to insure uniformity of the contamination level throughout the system; then eight 500 ml samples were withdrawn (one immediately after another) for particulate analysis.

The pressurized system consisted of a one-gallon mixing vessel, a magnetic stirring device to maintain a uniform suspension of particulate contaminant, associated plumbing, a low pressure air supply, and a sampling valve located near the outlet from the mixing vessel. Prior to each series of tests, the magnetic stirrer was operated for at least thirty minutes to insure uniform distribution of contamination in the mixing vessel; then the system was pressurized, and eight 500 ml samples were withdrawn for particulate analysis.

The fluid selected for the first series of samples with the laboratory recirculating system consisted of "as received" solvent grade trichloroethylene and was designated as Level 1 for this investigation. Except for No. 7, all of the remaining contamination levels were achieved by partial removal (by means of a wire mesh filter) of contaminant from the fluid recovered after the first series. After completion of the analysis of samples for the first six contamination levels, it was noted that an additional series of tests was needed to provide intermediate counts for the larger size ranges, and a second lot of fluid having particulate contaminant of similar origin and appearance was obtained.

All microscopic analyses were made in accordance with MSFC-PROC-166A "Procedure for Cleaning, Testing, and Handling of Onboard Hydraulic System Components and Hydraulic Fluids," except that sample volumes of 500 mls were used in place of the recommended volumes of 100 mls. For each group of eight samples, four were tested by each method; the following sequence was used to minimize possible effects due to changes in contamination levels during the sampling operation.

Sample Number	Analysis Method
1	Millipore
2	Whatman
3	Whatman
4	Millipore
5	Whatman
6	Millipore
7	Millipore
8	Whatman

The results are summarized in Table I.

DISCUSSION

Reproducibility of Results

Preliminary inspection of the data failed to indicate systematic changes in contamination levels during the various sampling operations. The counts for each method, therefore, were treated as replicate samples for the calculation of standard deviations. The results are summarized in Table I and presented in FIG 1.

The straight line drawn through the data was obtained previously in a more extensive study (Ref. 1) of the reproducibility of microscopic counts. Inspection of FIG 1 indicates that, in general, both the Millipore and Whatman values appear to be in agreement with the previous results (based only on Millipore data), the scatter about the line being attributable in large part to the smaller number of replicate samples (four) represented by each point.

As in previous investigations, these data indicate that the principal factor influencing the reproducibility of results is the number of particles counted, and other factors, such as size of particles and type of filter paper, exert relatively little influence.

Correlation of Whatman and Millipore Counts

Results shown in FIG 2 indicate that, for any size range, the Whatman and Millipore data can be approximated by a straight line passing through the origin, the scatter of data being only slightly greater than that which would be expected on the basis of normal experimental variations. Least square equations for the different ranges are given in Table II with other statistical parameters of interest. Taken together, these data indicate that the Whatman results correlate closely with the Millipore results, the slopes of the straight line equations for the different sizes ranging from approximately 12 to 3, the larger values being determined for the smaller size ranges.

Three possible explanations for the occurrence of slopes markedly greater than unity are immediately evident:

1. Migration of particles through the Whatman filter

2. Inability to distinguish particles retained on the Whatman filter surface

3. Embedding of particles in the Whatman filter.

Each of these mechanisms would be expected to be more important for the smaller particle sizes and, thus, are consistent with the observed slopes of the regression lines. A number of additional tests has been made in an attempt to assess the relative importance of these different mechanisms.

To determine if particulate material tends to migrate through the Whatman filter, a quantity of yellow plastic beads ranging from 5 to 150μ in size was introduced upstream of a Whatman filter, and the effluent from the downstream side of the filter was refiltered using a Millipore filter. Examination of the Millipore surface failed to reveal any of the colored plastic beads, thus indicating that migration of particulate contamination is unimportant for size ranges greater than 5μ. Similar results were obtained in a previous study (Ref. 2) using 20-40μ particles of silicon carbide as a source of distinguishable particles.

In considering the possibility that particulate contaminant retained on the Whatman filter surface may not be distinguishable as such, it must be recognized that the Whatman paper consists of a mat of fibers which afford a markedly nonuniform surface when viewed microscopically. The filtration characteristics of this material are derived from relatively long flow paths through successions of voids of irregular sizes and shapes. By way of contrast, the Millipore membrane affords almost complete retention of material larger than the rated pore size on the filter surface. It, therefore, would be expected that the slopes of the lines shown in FIG 2 would be influenced by the ease with which the contaminant particles can be distinguished from the fibers comprising the Whatman surface. To confirm this expectation, additional sets of eight samples each were obtained for two lots of fluid for which the contaminant particles were known to consist primarily of black O-ring material. The results, not given, indicated generally equal or higher Whatman counts for any given Millipore count than noted with the original test fluid.

The embedding of particulate material in the body of the Whatman filter paper was studied by several different methods. After the initial microscopic examination, a Whatman test filter was rendered partially transparent and was reexamined by an oil immersion technique. Although this treatment did not afford sufficient transparency to permit a total particulate count, it was sufficient to indicate numerous large particles embedded in the filter structure. This suggests that any numerical relation between results for these methods is limited in applicability to contaminant having characteristics very similar to that used as a basis for the evaluation. This is consistent with the results of a previous investigation (Ref. 3) in which contaminants from diverse origins were used and the correlation of results indicated an excessive amount of scatter.

As a second approach, two sets of test sample filtrates (one each for very high and very low contamination levels) were analyzed to determine the amount of non-volatile residue. The results (summarized in Table III) failed to indicate any significant differences between results for the two types of filters. The finding that the quantity of non-volatile contaminant passed by each type filter was approximately the same strongly suggests that the quantity of contaminant retained was almost the same.

Taken together, these results indicate that comparably rated Millipore and Whatman papers afford approximately equal retention of particles but that many particles retained by the Whatman filter are indistinguishable, either because their appearance is similar to that of the fibers comprising the Whatman surface or because they become embedded in the filter structure.

To obtain additional information about the embedding process, several Whatman test papers were examined microscopically; then they were returned to the filter holder and rinsed three times with 50 ml of clean solvent. Subsequent microscopic reexamination of the filters failed to indicate significant changes in the number of distinguishable particles. This suggests that whether or not a given particle becomes embedded in the filter structure or remains on the surface is strongly dependent on the exact location of its initial contact with the filter surface.

CONCLUSIONS

The reproducibility of results obtained by either the Whatman or Millipore method is determined by the number of particles counted. However, since a larger number of particles usually is counted by the Millipore method, results obtained by this method are more precise.

Although the Millipore and Whatman filters provide approximately equal retention of particles in the size range of interest, many particles retained by the Whatman filter are indistinguishable because either their appearance is similar to that of the fiber comprising the filter surface or they become embedded in the filter structure.

Numerical correlation of results by these methods, therefore, yields regression lines passing through the origin and having slopes deviating markedly from unity, the particular slope determined for any set of data being influenced by the appearance of the contaminant particles. It, therefore, appears that although specific relations may be developed for any particular contaminant source, no precise general correlation is possible.

TABLE I

SUMMARY OF MILLIPORE AND WHATMAN PARTICLE COUNT RESULTS

Size Range, Microns	10-25		25-50		50-100		100-175		175-700		> 700	
Analysis Method	Millipore	Whatman	Millipore	Whatman	Millipore	Whatman	Millipore	Whatman	Millipore	Whatman	Millipore	Whatman
Level #1	23,540 21,935 18,511 19,228	1,723 1,894 1,712 1,942	6,955 6,527 5,885 6,086	694 749 535 686	1,102 1,023 1,117 1,314	161 166 156 128	304 264 258 275	96 81 78 91	207 211 214 222	46 23 33 24	68 72 54 58	12 7 4 4
Mean Standard Deviation	20,803.5 2,345.7	1,817.7 117.5	6,363.2 477.0	666.0 91.7	1,139.0 123.7	152.7 17.0	275.2 20.4	86.5 8.4	213.5 6.4	31.5 11.0	63.0 8.4	6.8 3.8
Level #2	9,800 9,309 10,468 9,986	691 578 778 724	2,449 2,391 2,640 2,560	235 246 212 228	228 246 266 252	43 52 38 28	55 62 57 59	6 3 5 7	33 30 31 28	3 2 1 1	7 4 4 5	0 1 0 0
Mean Standard Deviation	9,890.8 479.2	692.8 84.5	2,510.0 111.5	230.2 14.2	248.0 15.7	40.3 10.0	58.3 3.0	5.2 1.7	30.5 2.1	1.7 1.0	5.0 1.4	0.2 0.5
Level #3	5,029 4,851 5,344 4,984	177 196 185 179	288 263 275 268	48 53 55 50	64 72 72 75	10 9 11 9	17 15 20 18	3 2 1 2	7 10 6 8	1 0 1 1	1 2 1 0	0 0 0 1
Mean Standard Deviation	5,052.0 208.8	184.2 8.5	273.5 10.8	51.5 3.1	69.8 4.8	9.8 1.0	17.5 2.1	2.0 0.8	7.8 1.7	0.7 0.5	1.0 0.8	0.2 0.5
Level #4	3,981 3,319 3,652 3,841	138 130 141 152	228 216 235 248	36 31 40 31	43 39 46 40	7 6 7 7	12 12 14 12	2 1 1 1	3 2 2 2	0 0 1 0	1 0 0 0	0 0 0 0
Mean Standard Deviation	3,698.2 286.5	140.2 9.1	231.7 13.4	34.5 4.4	42.0 3.2	6.8 0.5	12.7 1.0	1.2 0.5	2.2 0.5	0.2 0.5	0.2 0.5	0.0 0.0
Level #5	1,235 1,408 1,531 1,612	11 26 18 13	112 146 132 123	4 3 6 7	31 46 40 36	2 3 2 4	8 7 8 6	2 0 3 2	1 1 1 1	0 0 0 0	0 1 1 1	0 0 0 0
Mean Standard Deviation	1,446.5 164.1	17.0 6.7	128.2 14.2	5.0 1.8	38.2 6.3	2.7 1.0	7.0 0.8	1.7 1.3	1.0 0.0	0.0 0.0	0.7 0.5	0.0 0.0
Level #6	406 236 208 283	9 6 7 6	148 48 33 66	1 2 1 2	52 12 16 28	0 0 1 2	34 2 12 8	0 0 0 0	14 2 1 1	0 0 0 0	0 0 0 0	0 0 0 0
Mean Standard Deviation	283.2 87.5	7.0 1.4	73.8 51.3	1.5 0.6	27.0 18.0	0.7 1.0	14.0 14.0	0.0 0.0	4.5 6.4	0.0 0.0	0.0 0.0	0.0 0.0
Level #7	13,268 15,408 16,057 14,552	1,434 1,366 1,166 1,221	3,531 4,387 3,638 3,745	385 332 292 346	856 1,070 963 978	118 102 128 126	172 158 166 174	39 36 33 33	106 92 96 98	23 24 14 19	19 20 28 24	2 8 7 21
Mean Standard Deviation	14,821.1 1,205.0	1,296.7 124.5	3,825.2 384.5	338.7 38.4	966.7 87.7	118.5 11.8	167.5 7.2	35.2 2.9	98.0 5.9	20.0 4.5	22.8 4.1	9.5 8.1

TABLE II

EQUATIONS FROM POOLED DATA FOR MILLIPORE AND WHATMAN ANALYSES

<u>10-25μ Size Range</u>

$$MC = 11.8\ WC$$
$$S_{MC} = 1714 \text{ particles or } 21 \text{ percent}$$

<u>25-50μ Size Range</u>

$$MC = 10.0\ WC$$
$$S_{MC} = 259 \text{ particles or } 14 \text{ percent}$$

<u>50-100μ Size Range</u>

$$MC = 7.7\ WC$$
$$S_{MC} = 39 \text{ particles or } 11 \text{ percent}$$

<u>100-175μ Size Range</u>

$$MC = 3.4\ WC$$
$$S_{MC} = 28 \text{ particles or } 35 \text{ percent}$$

<u>175-700μ Size Range</u>

$$MC = 6.3\ WC$$
$$S_{MC} = 15 \text{ particles or } 30 \text{ percent}$$

<u>≥700μ Size Range</u>

$$MC = 4.7\ WC$$
$$S_{MC} = 16 \text{ particles or } 118 \text{ percent}$$

NOTE: MC = Millipore count, number of particles.

WC = Whatman count, number of particles.

S_{MC} = Standard error for the average of four Millipore counts calculated from the average of four Whatman counts.

TABLE III

SUMMARY OF NON-VOLATILE RESIDUE
DETERMINATIONS ON MILLIPORE AND WHATMAN SAMPLES

Values in Body of Table are NVR Contents of Filtrates, grams/500 ml								
Filter Type Used	Millipore	Whatman	Whatman	Millipore	Whatman	Millipore	Millipore	Whatman
High Contamination Level	0.3128	0.3021	0.2977	0.3066	0.3192	0.3135	0.3277	0.3181
Low Contamination Level	0.0011	0.0007	0.0005	0.0006	0.0007	0.0006	0.0009	0.0005

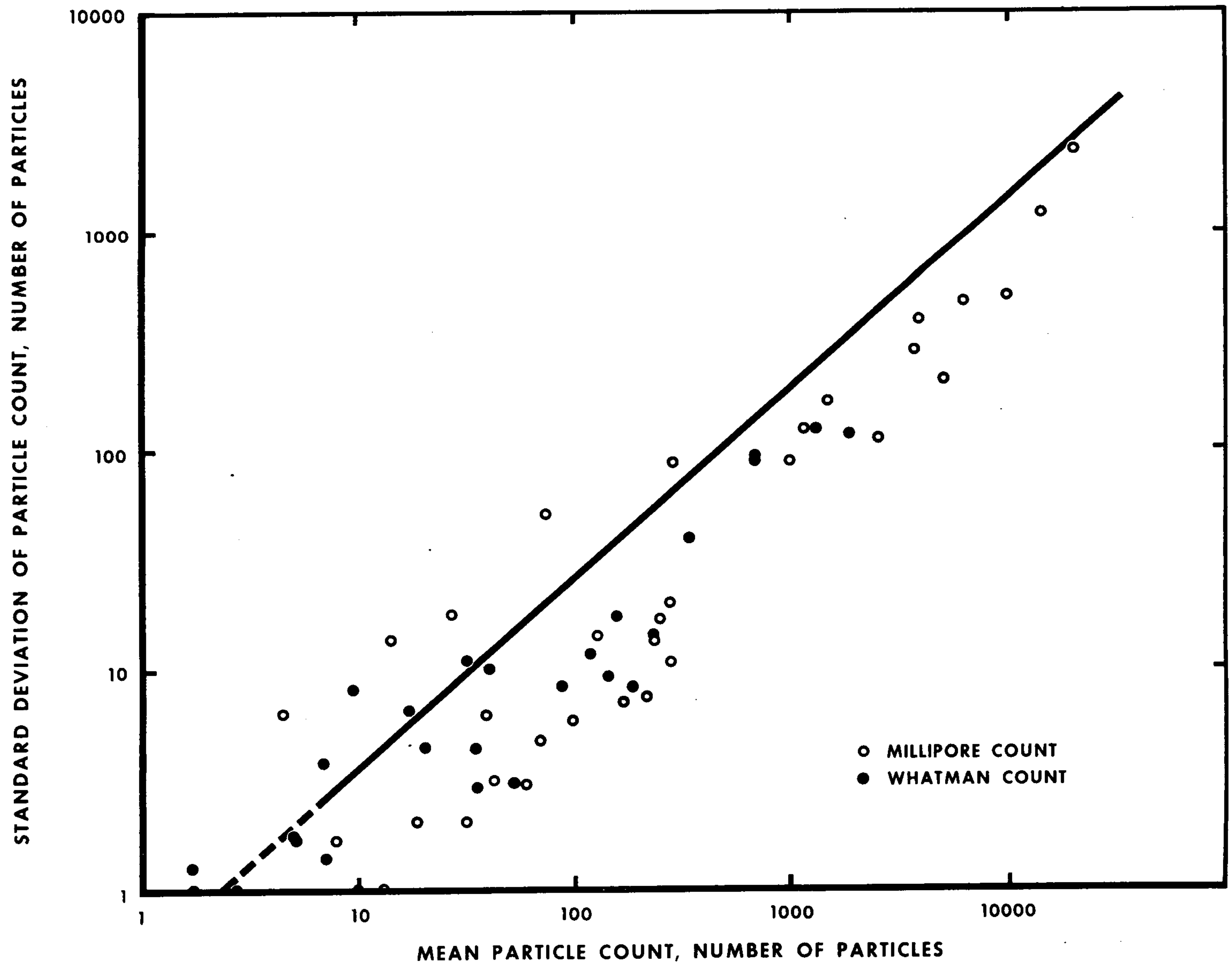

FIGURE 1 VARIATIONS IN STANDARD DEVIATIONS OF WHATMAN AND MILLIPORE COUNTS WITH NUMBER OF PARTICLES COUNTED

C-H 5855

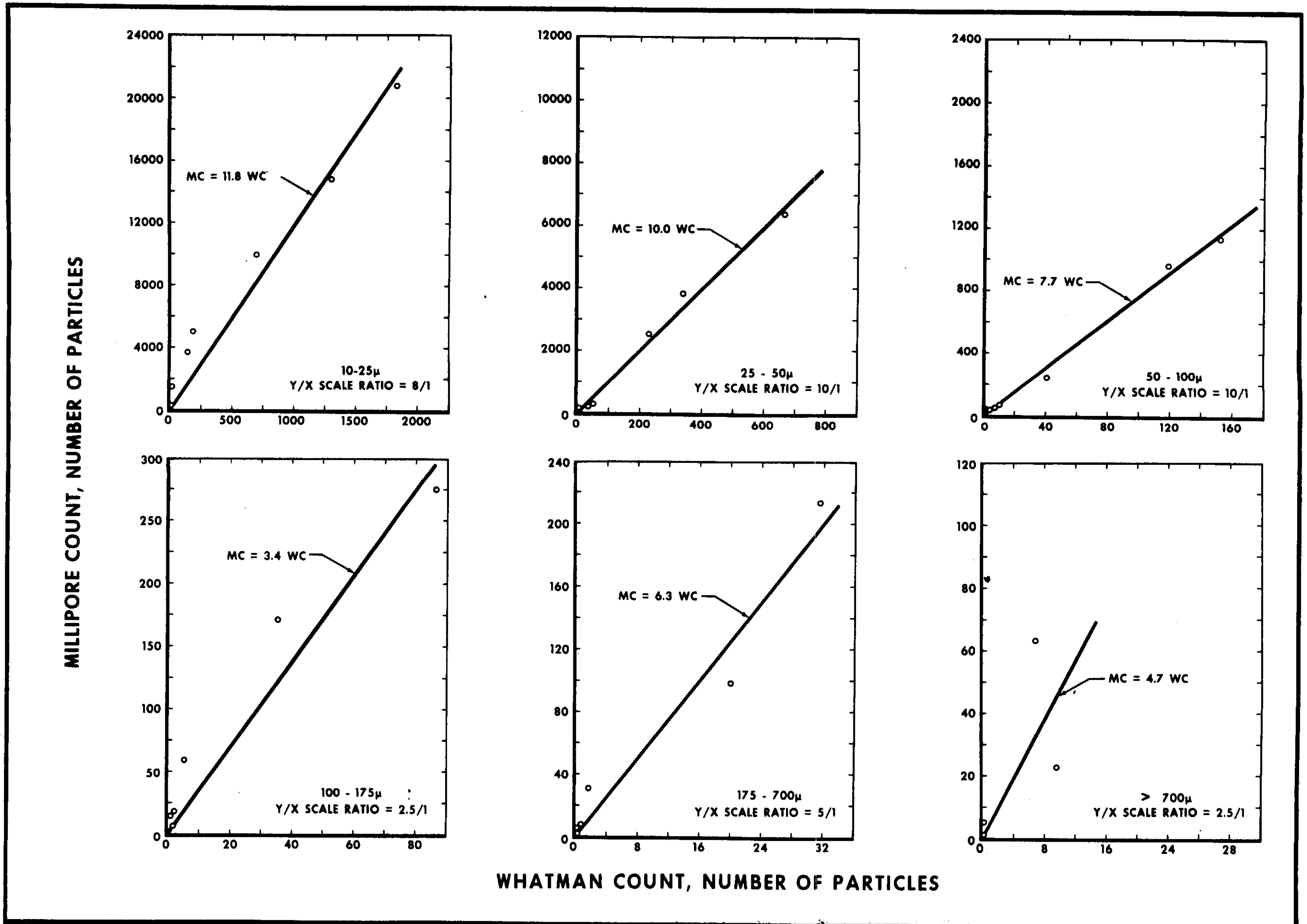

FIGURE 2 COMPARISON OF MILLIPORE AND WHATMAN PARTICLE COUNTS

C-H 5754

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